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D-STAR Technologies, Inc. has developed a technique for writing gratings through the polymer coating of commercially available optical fibers. In this Phase I project we discovered how to make our gratings thermally stable without destroying their polymer coating. This last discovery is an exciting breakthrough, for without it D-STAR's gratings would not have been commercially attractive. D-STAR now has the ability to both write *and* anneal gratings without removing their polymer coating. D-STAR's near-UV grating fabrication technique is a practical and inexpensive alternative for producing fiber gratings for diode laser stabilization, sensors and filters. We demonstrated that the concentration of germanium oxygen-deficient centers in the fiber core correlates well with the photosensitivity of fibers, and this concentration can vary by a factor of ~2 in supposedly identical fibers according to the detailed fiber preparation conditions. We showed that tin-doped fibers may prove useful for extending the response of hydrogen-loaded fibers into the near UV and visible. A plastic phase mask with a polymer film is feasible for fabrication of Bragg gratings using near UV light, but the absorption of the polymer film must be decreased to prevent its thermal damage.

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FINAL Progress Report: for the period 9/1/98 – 2/28/99

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Program Manager: Dr. Howard Schlossberg

Contract number: F49620-98-C-0076

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Personnel Supported: Dr. Dmitry Starodubov, Victor Grubsky

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Fabrication of Ultrastrong Fiber Bragg Gratings Through the Polymer Coating

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SUMMARY

During this Phase I project:

1. Using D-STAR Technologies, Inc.'s new near-UV writing technique we fabricated strong Bragg gratings (R > 70%) in commercially available fibers with NA=0.13 without stripping their polymer coating.

2. We invented a method to increase the amount of light delivered to the fiber core without damaging the polymer coating, thereby increasing the reflectivity of the Bragg gratings.

3. We discovered a simple way to stabilize the gratings without harming their polymer coating. (Very important!)

4. We tested the sensitivity of various photosensitive fibers in the near-UV spectral region.

5. We performed preliminary tests of alternative photosensitive materials.

6. We tested a novel technology of using *plastic* phase masks to write fiber Bragg gratings.

WHAT THE MARKET WANTS:

a) **For stabilizing 980 pump lasers:** inexpensive Bragg gratings with reflectivity R ~ 2% and high mechanical strength written in fibers of numerical aperture NA≈ 0.13.

b) **For end mirrors for fiber lasers:** Bragg gratings with R~50% – 80%.

c) **For fiber sensors:** narrow Bragg gratings with R~50% – 80%.

1) WRITING EFFICIENT BRAGG GRATINGS THROUGH THE POLYMER COATING OF OPTICAL FIBERS

D-STAR's goal is to develop an inexpensive technology for writing Bragg gratings in commercially available optical fibers. Bragg gratings are imprinted into optical fibers by exposing the fiber from the side to patterned UV light. Most commercial Bragg gratings are fabricated using mid-UV light at 244 - 248 nm or 193 nm. Because the acrylic coatings of optical fibers are opaque to UV light at these wavelengths, the fiber must first be stripped of its coating to imprint the grating, and then recoated afterwards. Stripping the fiber and then recoating it mechanically weakens the fiber and makes grating fabrication cumbersome and expensive.

We recently demonstrated Bragg grating fabrication using near-UV light at ~330 nm [1]. Because many fiber coatings are partially transmissive at 330 nm, with near-UV light one can fabricate Bragg gratings right through the fiber coating [2]. (In contrast, Lucent Technologies is developing special coatings that are transmissive in the mid-UV, at 240 nm. [3]) In our previous experiments we fabricated Bragg gratings with moderate reflectivity (R~20%) in an acrylate-coated fiber [2]. Using hydrogen-loaded fibers with high germanium content and a silicone coating transparent at 300 nm, we were able to fabricate high-reflectivity (R = 97%) Bragg gratings [4].

In this Phase I project we developed a practical system for writing Bragg gratings in *commercially available, low-numerical-aperture photosensitive fibers with standard polymer coatings and without hydrogen loading.* The fibers should have a small numerical aperture to minimize loss when the grating is spliced to ordinary telecommunication fiber (NA ≈ 0.13). We prefer to avoid hydrogen loading of the fibers because that process is time-consuming. We want to use commercially available fibers because we do not yet have the leverage or financial resources to dictate the compositions of fibers made by fiber manufacturers.

Using a specially designed UV fiber-optic spectrometer we analyzed the absorption of fiber coatings. We found that the most common acrylate fiber coatings transmit sufficient near-UV light to allow grating fabrication through the coating. These acrylate coatings are also sufficiently uniform so that they do not distort the transmitted near-UV wavefront.

To determine the optimum writing wavelength, we fabricated gratings using the near-UV argon ion laser lines at 334 nm, 351 nm, or 364 nm. Figure 1 shows that the acrylate polymer transmits roughly 42% at 334 nm, 61% at 351 nm, and 75% at 364 nm. Although the polymer

transmits more light at the longest UV wavelength, the photosensitivity of germanium-doped fibers in the near-UV band peaks at 330 nm and decreases for longer UV wavelengths.
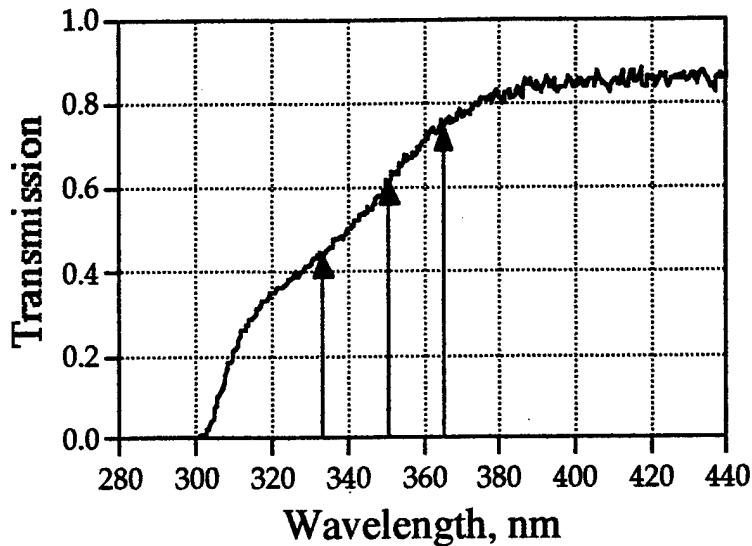

Fig. 1. Transmission of acrylate fiber coating available on photosensitive fibers from Fibercore Ltd. (UK).

An important factor to consider in choosing the writing wavelength is that UV irradiation increases a polymer's UV absorption. This increased absorption causes of the polymer to heat more rapidly, which in turn hastens its degradation. Therefore, photo-induced decay of the polymer coating limits the possible grating writing time. We were able to solve this problem, as we show below.

Figures 2 and 3 show how the wavelength and intensity of the UV irradiation affects the polymer coating's degradation time. The polymer was irradiated until it cooked. Note that the longer wavelengths damage the polymer least.

To find the optimum UV wavelength for writing gratings through the polymer, we need to normalize the UV fluence by the fiber's photosensitivity. In a separate experiment we found that with our photosensitive fibers (PS1500 from Fibercore Ltd.) The ratio of photosensitivities at 334, 351 and 364 nm was 1 : (1/2.5) : (1/23). Using these factors, the data for polymer coating degradation with normalized UV fluences are shown on Fig. 3. Clearly, 351 nm is the best wavelength for writing through the polymer coating in this fiber. At 351 nm the polymer has a
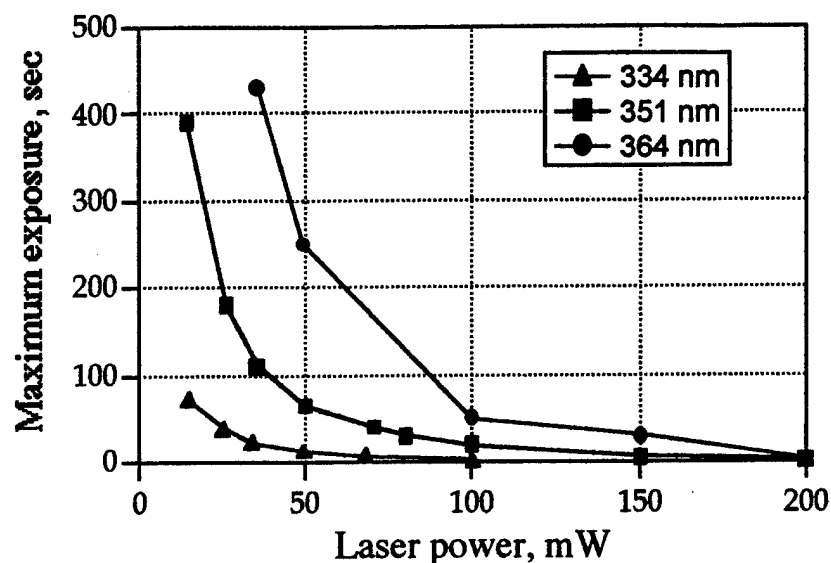
Fig. 2. Maximum exposure time tolerated by a polymer coating vs. UV power. Note that at the higher power the coating withstands less total fluence.

high transparency and a small rate of UV-induced degradation, while the fiber core remains reasonably photosensitive. The other two wavelengths (334 nm and 364 nm) can both write gratings in the core, but 364 nm takes 23 times longer than 334 nm, so it is not practical. Light at 334 nm may be practical for weak gratings because it writes faster than 351 nm, but for strong gratings it burns the polymer too quickly. In our subsequent experiments we used both 334 and
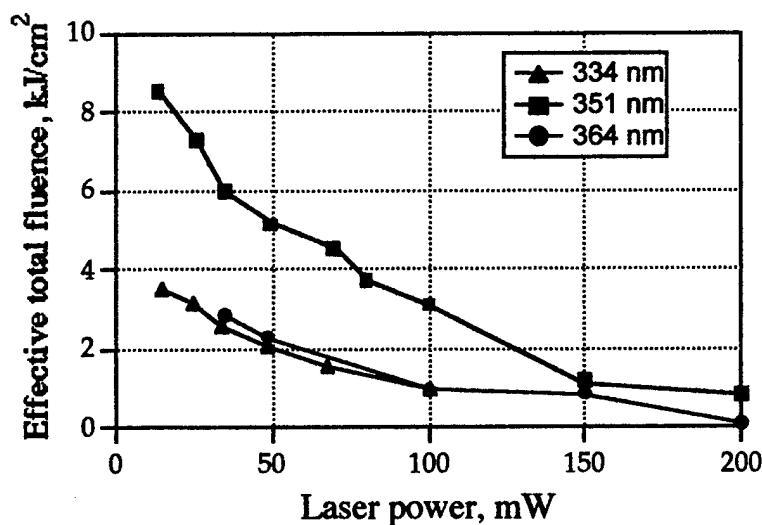

Fig. 3. Maximum UV fluence until coating cooked normalized by the photosensitivity of the core.

351 nm light for grating fabrication.

We found that by using boron/germanium-codoped fibers we could write efficient gratings with near-UV light through their acrylic polymer coating. Figure 4 demonstrates a typical transmission spectrum of a 1-cm long grating fabricated through the fiber polymer coating. The reflectivity of this grating was R = 67%. We are working to increase the reflectivity to R~80 - 90%.
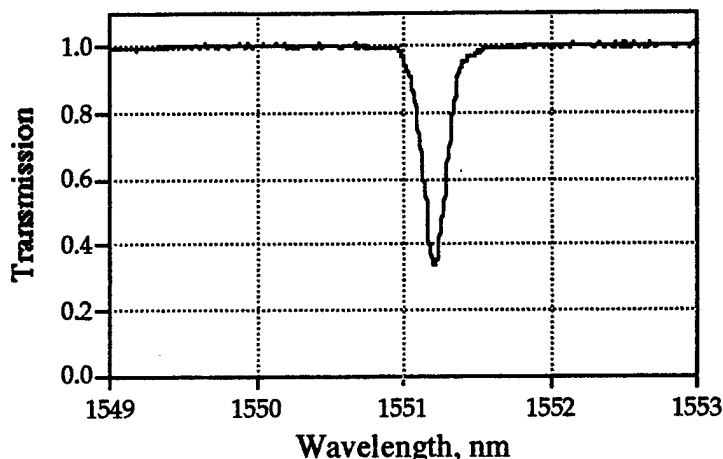
Fig. 4. Spectrum of a Bragg grating written in PS1500 fiber through its polymer coating using 334 nm light. Grating length is 1 cm.

2) MULTI-ANGLE WRITING DELIVERS MORE ENERGY TO THE FIBER CORE WITHOUT DAMAGING THE POLYMER COATING.

We produced the grating in Figure 4 using a simple, multi-angle writing technique. This reduces the UV fluence delivered to the polymer coating while preserving the fluence delivered to the fiber's core. The conventional writing technique focuses a narrow laser beam onto the fiber core with a cylindrical lens of small numerical aperture (NA ~ 0.05), as shown in Fig 5. Therefore, before getting to the fiber core, the focused UV beam passes through a very narrow section of the polymer coating. This produces a high power density within the polymer layer and damages it.

Our idea was to reduce the power density on the polymer coating by using a laser beam with a 10 times larger numerical aperture. This reduces the polymer degradation rate and so increases the time window available for writing gratings. One could simply focus a broad laser beam into a ~10 micron spot exactly at the core, but simple cylindrical lenses cannot do this;

they have too much aberration. We solved this problem by using a narrow laser beam but displacing the beam between consecutive exposures, each time re-adjusting the focus, as shown in Fig. 5. Using this method, we increased the maximum fiber exposure by a factor of 5 and produced gratings with much higher reflectivity. We are working on improving this technique by using better optical elements that have smaller residual aberrations.
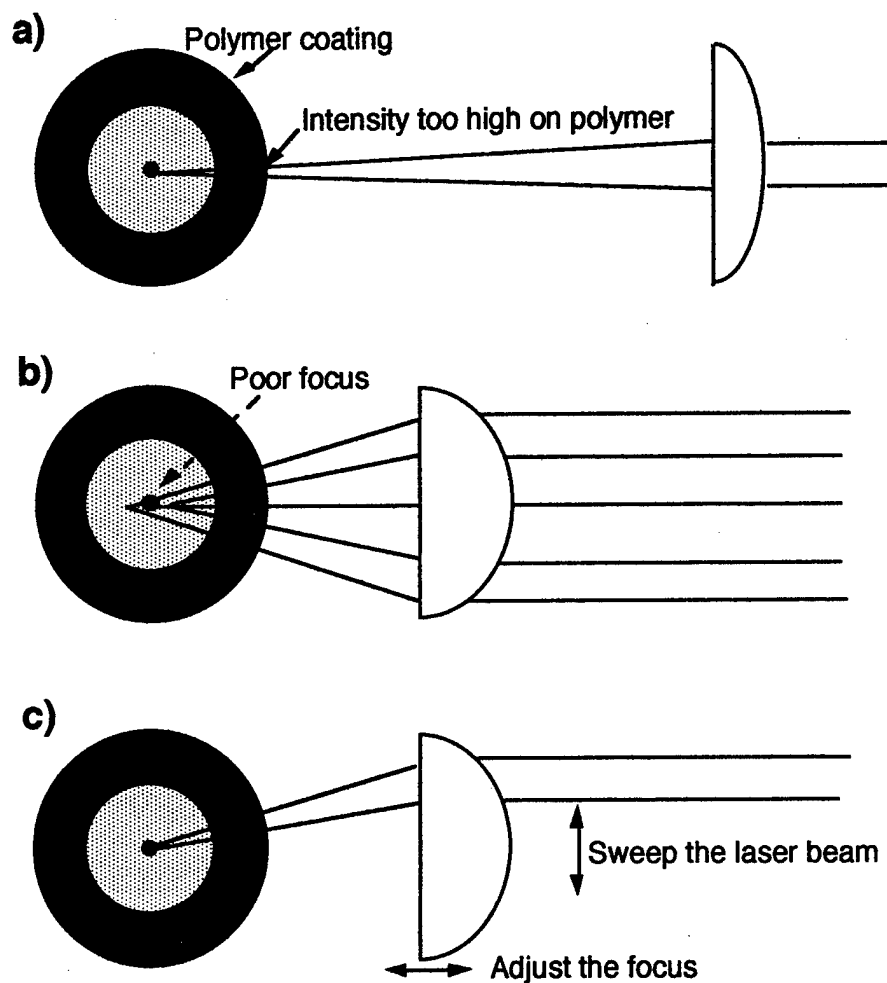

Fig. 5. New writing technique to increase the fluence delivered to the fiber core without destroying the polymer coating. a) Conventional writing uses a small NA, long focal length lens. b) A high NA, short focal length lens spreads the laser across the polymer and so reduces the intensity at the polymer, but the focal spot degrades. c) Sweeping a laser beam across the lens while adjusting the focus distributes the fluence over a large area of the polymer yet maintains a high intensity on the core. The focus adjustment is computer controlled.

3) STABILIZATION OF GRATINGS WRITTEN THROUGH THE POLYMER COATING

Bragg fiber gratings must last for decades with no significant loss of reflectivity. If not annealed, the reflectivity of a freshly written grating will degrade over time. To stabilize the grating's reflectivity the grating is usually annealed at elevated temperature (usually 200-300 °C) in order to ensure its stability under normal operating temperatures. We developed a technique that allows us to anneal fiber gratings using light and with the polymer in place, as described below.

2A) Effect of writing wavelength on temperature stability of fiber gratings

We first checked to see if gratings written with different near-UV wavelengths differ in their thermal stability. We fabricated three Bragg gratings in the same piece of fiber (PS1500) using three different near-UV wavelengths: 334, 351, and 364 nm. We then heated these gratings in the same stove at 200 °C. Figure 6 shows that all three grating suffered the same fractional decrease of their grating strength. Therefore, we concluded that gratings written with different UV wavelengths all have the same thermal stability.
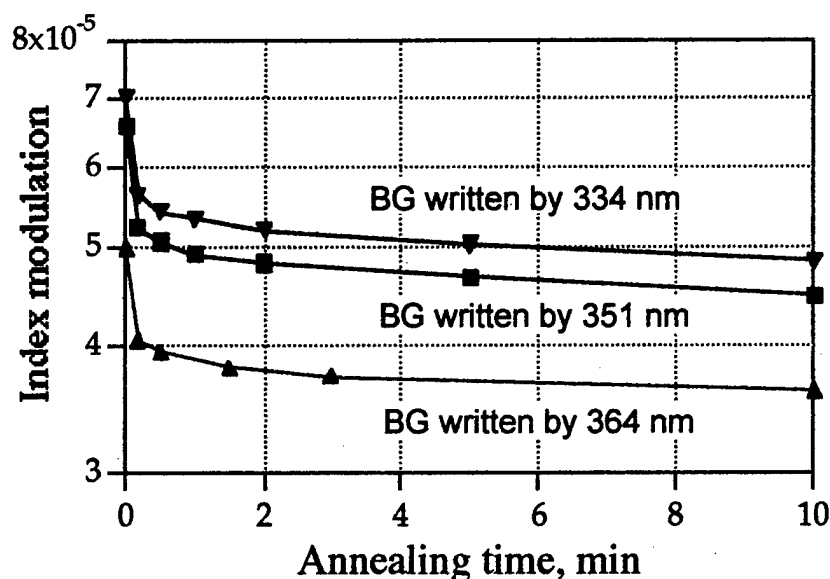

Fig. 6. Annealing of fiber Bragg gratings written with different near-UV wavelengths at 200°C

3B) Annealing with light instead of with heat

In our previous experiments we found that we could anneal gratings not by heat but by exposing them to a *uniform* UV light beam either before, during, or after grating fabrication. We attributed this improved stability fact to "burning out" the most unstable defects (germanium oxygen-deficient centers) with uniform UV exposure.

Initially we thought that this optical annealing technique might prove useful for our polymer-coated gratings because it could eliminate the need for thermal annealing, which we believed would destroy the fiber's polymer coating. However, with optical annealing the polymer's absorption once again limits the total UV fluence. The best wavelength for UV annealing through a polymer coating is 351 nm, since it allows the maximum exposure, as explained above.

Here we studied how post-exposing gratings with different wavelengths affects the grating's thermal stability. We fabricated three identical gratings in different locations of the same fiber using 334-nm light. We then post-exposed the three gratings with uniform beams of 300, 334, and 364-nm light respectively, until a significant portion of the index modulation was erased. We found that upon heating these gratings to 200 °C, all of the post-exposed gratings had more stability than a grating with no post exposure.

We also explored the novel idea of annealing with visible light launched into the fiber core instead of through the polymer coating. The fiber core's absorption is too large in the UV to allow UV light to travel more than a few mm along the fiber, but the fiber is relatively transparent at the 488 nm argon laser line. In principle 488 nm light could anneal the grating by using two-photon absorption into the 240-nm band of the core's germanium-oxygen deficient centers. However, because the 480-nm wavelength depends on two-photon absorption, it also requires a high power density.

Figure 7 shows our results using either near-UV or blue light to anneal gratings in a high-NA photosensitive fiber (PFBG-1355T from QPS Tech., Inc.). The gratings were post-exposed by a 488-nm beam through the core or by a uniform 351-nm UV beam from the side. Both of these samples showed improved thermal stability when heated to 200 °C compared to gratings that had no post exposure. However, post-exposures with blue or near-UV light were never

sufficient to make the gratings completely stable. Additionally, post-exposure with blue light took a long time (20 minutes to 1 hour). Similar treatments of gratings written in low-NA photosensitive fibers showed even less improvement in their stability. From this we conclude that UV annealing is more practical than annealing with visible light.
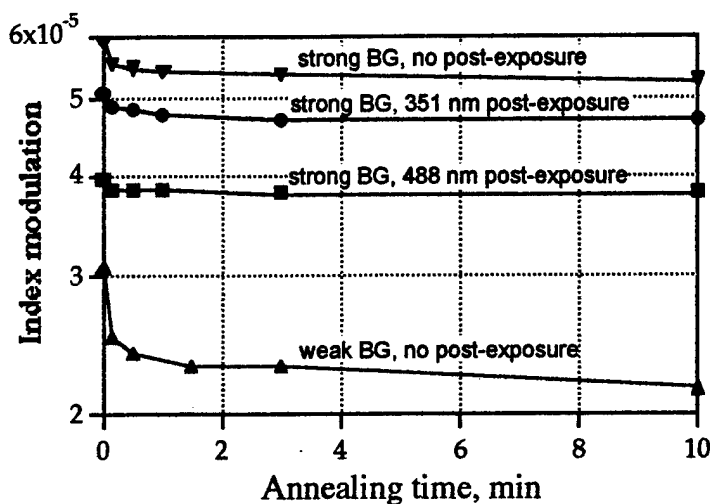

Fig. 7. Decay of Bragg gratings annealed with near-UV light or visible light.

4) FINDING THE MOST NEAR-UV SENSITIVE FIBER

D-STAR Technologies, Inc.'s goal is to develop its novel technology for writing Bragg gratings in optical fibers using near-UV light. We previously found that fibers co-doped with germanium and boron exhibit higher sensitivity to near-UV light than fibers doped only with germanium. Boron softens the glass and facilitates its structural transformation under UV irradiation.[1] Therefore, in this study we concentrated our efforts on boron co-doped fibers.

We obtained a number of fiber samples from Fibercore Ltd. (Southampton, UK). All these fibers have a high germanium concentration (~20 mol. %) and a significant amount of boron. Co-coping with boron serves two functions: it increases the photosensitivity of the fibers and it reduces their numerical aperture to 0.13, making the fibers splice with less loss to standard communication fibers.
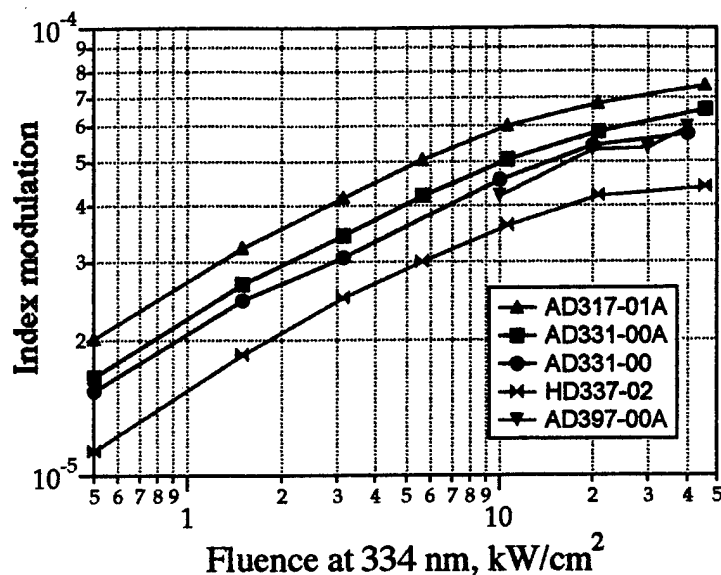
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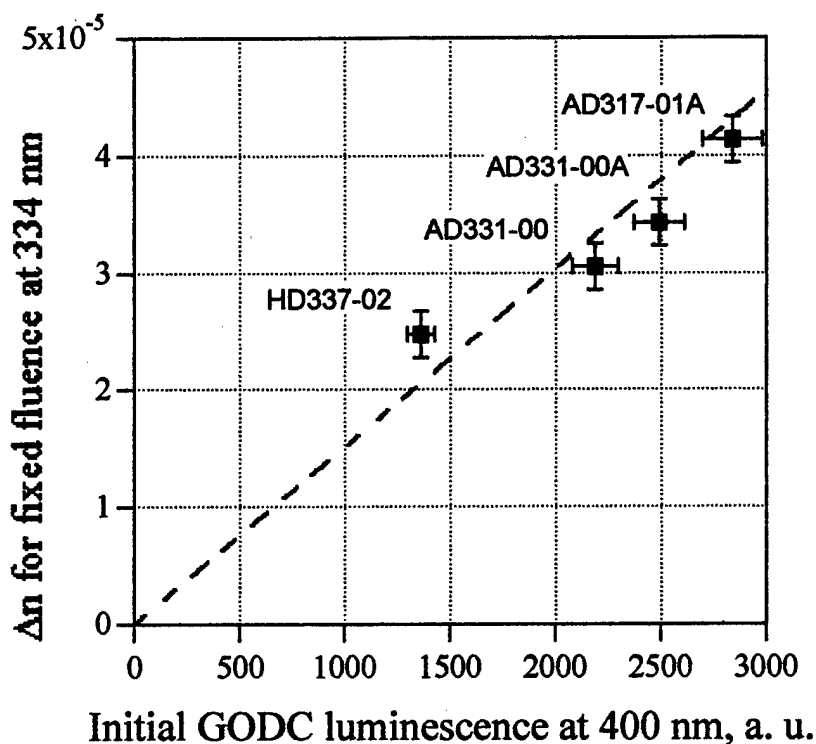Fig. 8. Variation in photosensitivity of B-doped fibers from Fibercore Ltd.

Fig. 9. Change in refractive index vs. concentration of defect centers in four fibers made by Fibercore Ltd. and having identical core sizes. The fibers were all irradiated with the same fluence of 334-nm light. These fibers were supposedly identical in composition, but, in fact, contained different concentrations of Germanium Oxygen Deficient Centers.

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In order to test the photosensitivity of these boron co-doped germanosilicate fibers, we stripped their coating and then irradiated them with 334-nm UV light. Although the manufacturer specified that all of these fibers have the same compositions, we found a significant difference in their photosensitivity (Fig. 8). In particular, we observed the largest photosensitive response in fiber #AD317-01A, which requires half the UV fluence to produce the same index change compared to the next best fiber, #AD331-00A. It is possible that fiber drawing conditions such as temperature and tension affected the photosensitivity.

We know from our previous experiments that the photosensitivity of Ge-doped fibers (with no hydrogen) is determined primarily by the initial concentration of germanium oxygen-deficient centers. To check if these fibers had different concentration of these defects, we illuminated fresh fiber samples with UV light and observed their fluorescence at 400 nm. Germanium oxygen-deficient centers have a strong emission peaked at this wavelength. Figure 9 shows a good correlation between the measured GODC concentration and the photosensitivity of the samples, indicating that the various fiber samples did indeed contain different concentrations of defects. The defect concentration in the fiber core is determined both by the preform fabrication technique and also by the pulling speed and drawing temperature. We have contacted Fibercore Ltd. and asked them to investigate the differences in the fiber manufacturing conditions of these samples, and we are working with them to produce even more photosensitive fibers.

5) TESTS OF ALTERNATIVE PHOTOSENSITIVE MATERIALS

In recent years a number of dopants have been used to make fibers photosensitive. Along with boron, a promising dopant is tin. Tin-doped fibers have low absorption losses in the 1.5 μm telecommunications spectral region. Gratings produced in these fibers have higher thermal stability compared to germanium/boron-doped fibers.[5] Tin and germanium have similar electronic structure and absorption bands, so we expect that tin-doped fibers may also be sensitive in near-UV region. Experimental data by Skuja [6] suggest that the excitation spectrum of tin oxygen-deficient centers extends further into the long-wavelength region than that of germanium centers. This implies that one can use longer wavelength light to excite these centers compared to the usual germanium oxygen-deficient centers. In tin-doped fibers we expect an

increase in photosensitivity at the 351 nm and 364 nm lines of the argon laser. When writing gratings through the fiber coating these longer wavelengths will prolong the life of the plastic coating surrounding the fiber.

We took preliminary measurements on a proprietary fiber from Southampton University (England) that was doped with both tin and germanium. Because the fiber had ~0.5 mol.% of tin and several mol.% of germanium, it was difficult to precisely determine the contribution of tin to the overall index change upon UV irradiation. We compared the relative change of photosensitivity with writing UV wavelength (Table 1). We used higher intensities at longer wavelengths in order to compensate for the decreased absorption. The data show that while in a germanium/boron co-doped fiber the index change decreased somewhat at longer wavelengths, in the germanium/tin co-doped fiber the index change increased at the longer wavelengths.

UV power and wavelength	Index change in $B_2O_3/GeO_2/SiO_2$ fiber	Index change in $SnO_2/GeO_2/SiO_2$ fiber
334 nm, 100 mW	7.2×10^{-5}	1.9×10^{-5}
351 nm, 200 mW	7.0×10^{-5}	2.2×10^{-5}
364 nm, 750 mW	5.3×10^{-5}	2.6×10^{-5}

Table 1. Comparison of photosensitivity of boron and tin co-doped fibers at different near-UV wavelengths.

The overall amplitude of the index change in the tin-doped fiber is smaller due to the smaller concentrations of tin and germanium. However, these results suggest that a fiber with a tin and possible boron co-doping could be a promising material for writing gratings through the fiber coating using 351 or 364 nm laser lines.

6) HYDROGEN-LOADED FIBERS

D-STAR Technologies, Inc. is also working to increase the photosensitivity of hydrogen-loaded fibers. Loading the fiber with molecular hydrogen completely changes the chemistry of the reaction induced by light. Without hydrogen it is the germanium *defects* that determine the photosensitivity of the fiber. However, if hydrogen is present then all of the germanium atoms (and not only their defects) participate in grating formation. Our experiments indicate that in the presence of H_2, UV light breaks the Ge-O bond and a nearby hydrogen molecule subsequently splits to form O-H, and, to a lesser extent, other Ge-related defects. This implies that the photosensitivity of a hydrogen-loaded fiber no longer follows the absorption spectrum of Ge defects, but instead follows the absorption spectrum of the Ge-O bond, which peaks far in the UV, at $\lambda < 200$ nm. Consequently, in hydrogen-loaded fibers the photosensitivity increases monotonically as the wavelength approaches 200 nm.

In order to write gratings efficiently through the polymer coating of a hydrogen-loaded fiber the coating must be designed to transmit deep into the UV. An alternate approach is to dope the glass with a material other than germanium and with a fundamental absorption in the near-UV. We are testing glasses that have structure similar to germanosilica, and especially titanium-doped silica, which has an absorption edge at ~300 nm (Fig. 10). Although in our preliminary experiments we were unable to detect photoinduced changes in this material with hydrogen loading, we will repeat these experiments.
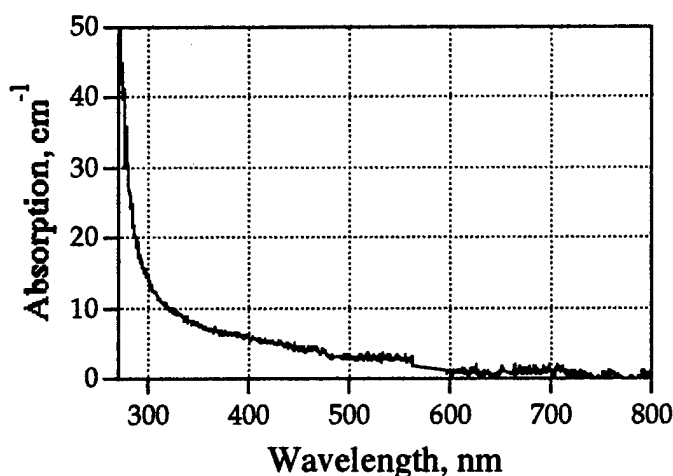

Fig. 10. Absorption spectrum of titanium-doped silica glass. Note the long tail into the near-UV and visible wavelengths.

7) WRITING FIBER GRATINGS USING PLASTIC PHASE MASKS

Commercially available silica masks are expensive (~$5,000) because they are produced by electron beam lithography and ion etching. Our success with writing gratings through the polymer coating of fibers suggests that we can use plastic phase masks instead of commercial silica phase masks. Plastic phase masks can be mass-produced at very little cost (~$50). It may even be possible to imprint the phase mask into the fiber coating itself, so that the fiber serves as its own phase mask.

A commercially useful phase mask made from plastic must show little degradation under UV exposure. We contacted Wavefront Technology Inc. of Paramount, CA to help with our development and testing of plastic replica masks. We first tested thin planar films of a polymer supplied by Wavefront Technology. In our first set of experiments we measured the transparency and light stability of replicable polymers, including a standard acrylic film on a PMMA substrate, as shown in Fig. 11. This sample was transparent to 330-nm light (which we use to write gratings in fibers without hydrogen) and to 300-nm light (which we use for hydrogen-loaded fibers).
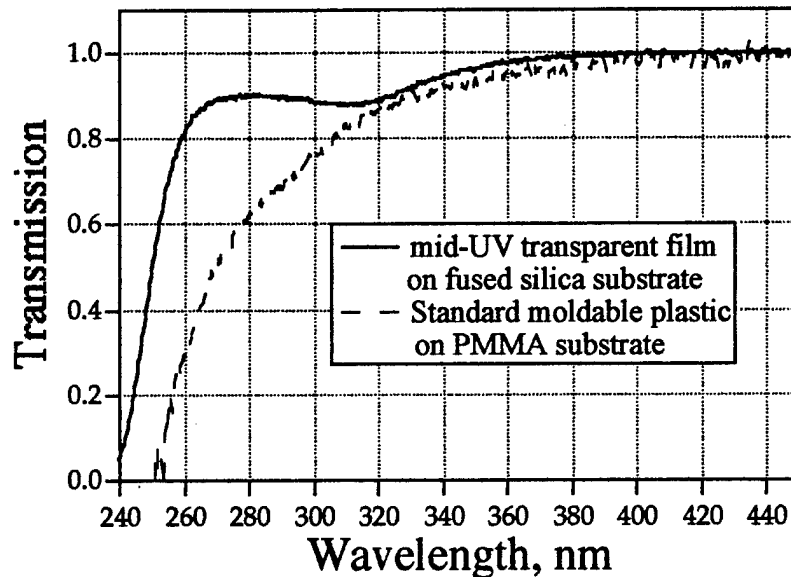

Fig. 11. Transmission spectra of moldable polymer films: (a) (dashed line) standard moldable acrylic plastic on polymethylmethacrylate substrate; (b) special UV transparent polymer film on UV-grade fused silica substrate.

We then measured how long the acrylic film could survive under the intense UV exposure typically used for grating fabrication. The polymer film was ~5 micron thick and was exposed with a UV beam focused by a cylindrical lens. Figure 12 shows that the lifetime of the film decreased with shorter wavelengths. However, the films were not stable enough to allow fabrication of more than ~10 gratings. The principal damage mechanism was formation of bubbles and separation of the film from the substrate.

We then had Wavefront Technology fabricate a more transparent UV polymer and use silica glass as the substrate. This film was much thicker, ~130 μm, and was transparent for wavelengths $\lambda > 250$ nm. Now the damage mechanism was different; the film merely darkened and did not form bubbles. However this thick film showed lower resistance to radiation than the first film; in our experiments at 330 nm the time to burn was reduced by a factor of ~3. Wavefront Technology is preparing a set of thinner films on silica substrates, and we expect that these films will have longer lifetimes. Wavefront Technology did make 10 replicas of silica phase masks out of acrylic plastic on PMMA. All replicas had very high optical quality and showed uniformity of diffraction across the mask. The amount of light in 0th order was suitably
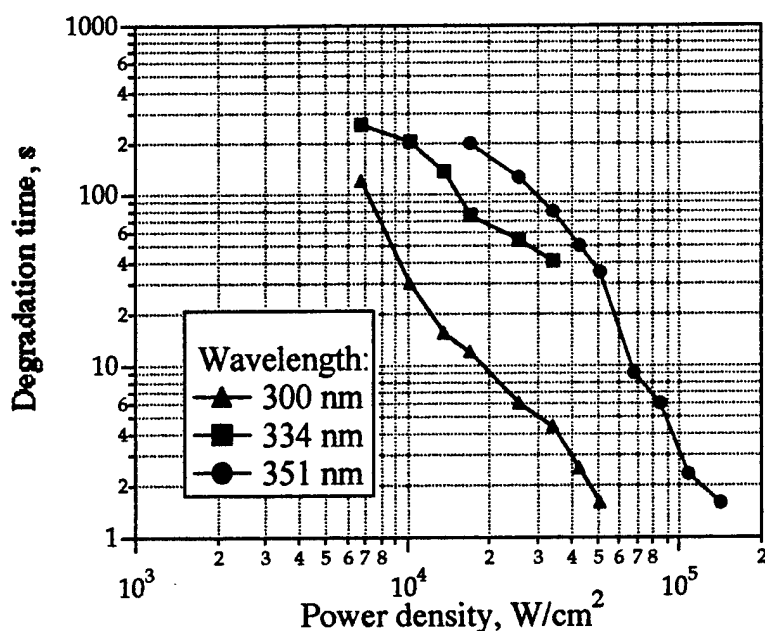

Fig. 12. Burn tests for standard moldable polymer film. The films must withstand a power density of 10^4 W/cm^2 for a typical one-grating writing time of 100s. The polymer coating of a fiber must withstand this exposure once; a practical plastic phase mask should withstand 100 to 10,000 such exposures.

small: only ~12% for 330 nm and 19% for 364 nm. All the masks had quality suitable for grating writing.

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9) CONCLUSIONS

D-STAR Technologies, Inc. has developed a technique for writing gratings through the polymer coating of commercially available optical fibers. In this Phase I project we discovered how to make our gratings thermally stable without destroying their polymer coating. This last discovery is an exciting breakthrough, for without it D-STAR's gratings would not have been commercially attractive. D-STAR now has the ability to both write *and* anneal gratings without removing their polymer coating. D-STAR's near-UV grating fabrication technique is a practical and inexpensive alternative for producing fiber gratings for diode laser stabilization, sensors and filters. We demonstrated that the concentration of germanium oxygen-deficient centers in the fiber core correlates well with the photosensitivity of fibers, and this concentration can vary by a factor of ~2 in supposedly identical fibers according to the detailed fiber preparation conditions. We showed that tin-doped fibers may prove useful for extending the response of hydrogen-loaded fibers into the near UV and visible. A plastic phase mask with a polymer film is feasible for fabrication of Bragg gratings using near UV light, but the absorption of the polymer film must be decreased to prevent its thermal damage.